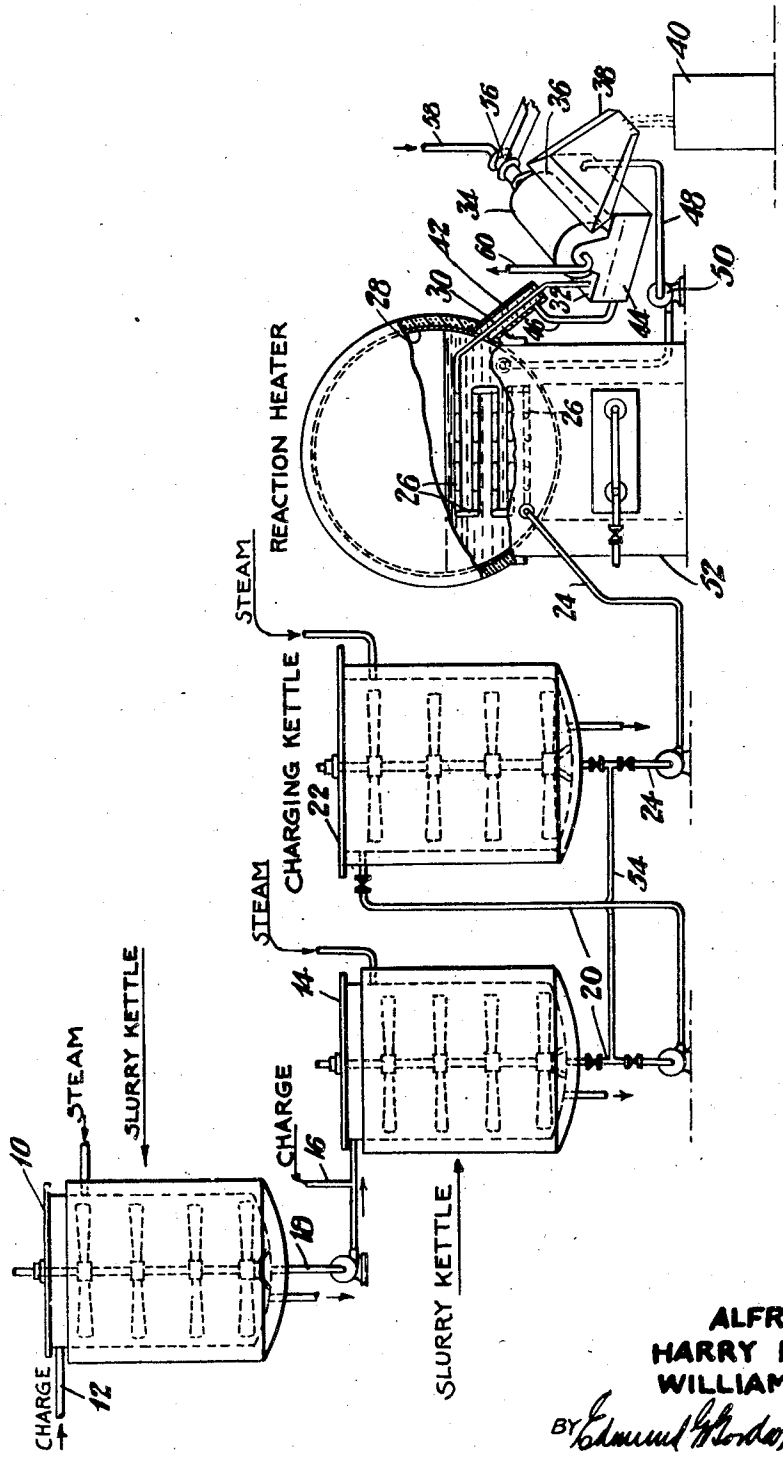
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PROCESS FOR MANUFACTURING LITHIUM BASE GREASES

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PROCESS FOR MANUFACTURING LITHIUM BASE GREASES

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This invention relates to improvements in the manufacture of greases and more particularly to the manufacture of lithium base greases.

Lithium base greases are of particular interest because lithium soaps give greases which have good lubricating properties at extremely low temperatures of the order of minus 100° F. At the same time the greases made from lithium soaps are also useful at temperatures of the order of approximately 300° F. This wide range of utility is especially advantageous in the lubrication of aircraft and other equipment which must be operated at ordinary temperatures as well as at extremely low temperatures at which other greases become stiff and hard and fail to lubricate.

In the manufacture of lithium base greases a number of difficulties have been encountered which are not common to the manufacture of sodium and other greases. For example, no way has been developed for the manufacture of a lithium base grease with Pennsylvania type oils without the use of other materials. The reason for this is not understood since satisfactory greases have been made with Coastal type oils of a wide range of viscosity. Furthermore, some of the greases made with Pennsylvania oils, lithium soap and stearic acid, for example, were not water-repellent, whereas the grease made with a Coastal oil and a lithium soap was water-repellent. Other difficulties have been encountered in the heating of the grease mixture, since excess or prolonged heating and excessive agitation at high temperatures, destroy the tackiness and water-repellent properties of the grease. The maintenance of the grease at the maximum heating temperature for an appreciable length of time destroys its grease-like properties.

The primary object of the present invention is to provide improved method of manufacturing greases by which various factors are controlled and coordinated to produce a satisfactory grease.

A further object of the invention is to provide an improved process for the successful manufacture of lithium base greases and which overcomes the various special difficulties encountered in the use of lithium soaps.

According to the present invention the improved process in general includes the heating and mixing of the lithium soap with a portion of the oil to be used in making the grease, the separate heating and mixing of an aluminum soap in another portion of the oil, mixing these materials and adding to the resulting mixture any other ingredients desired in the final grease such as an antioxidant, an elastic polymer, an E. P. agent,

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etc., passing the resulting mixture in a confined stream of narrow cross-section and of great length through a heating zone in which the mixture is raised to a final temperature of from about 400° F. to about 450° F., depending on type of final product desired, maintaining the material at the final temperature for not more than a few minutes, and then quickly cooling the grease in thin layers down to ordinary temperatures.

The improved process of the present invention also includes other features such as the simultaneous working and heating of the grease mixture in the confined stream of restricted cross section.

The present invention, together with its various features and advantages, will be apparent to those skilled in the art from the following more detailed description thereof, taken in connection with the accompanying drawing and examples.

In the drawing:

The single figure is a diagrammatic view showing an apparatus particularly adapted for carrying out the improved process.

Referring to the drawing, a preferred form of the improved process of the present invention will be illustrated in connection with the manufacture of a batch of 4,000 pounds of a lithium base grease from 600 steam refined Pennsylvania lubricating stock. The constituents going into this batch of grease comprised 458 gallons of the lubricating oil stock, 88 pounds of aluminum tristearate, 440 pounds of lithium stearate, 40 pounds of polyisobutene having a molecular weight of about 20,000, and then pounds of tributyl phosphite as an oxidation inhibitor. The process is started by charging the 440 pounds of lithium stearate and 40 gallons of the oil into a steam jacketed mixing kettle 10 in which these ingredients are simultaneously mixed and heated to a temperature of 150°–200° F., and preferably to about 170° F. The oil may be run into the kettle 10 through a feed line 12 while the lithium stearate is dumped in by any suitable means. At the same time 229 gallons of the oil and the 88 pounds of aluminum tristearate is introduced into a second steam jacketed kettle 14, the oil being run in through a supply line 16. The mixture in the kettle 14 is agitated thoroughly while heating it to a temperature of 270° F. by means of the steam jacket. After this temperature is reached in the kettle 14, the remaining oil, 189 gallons, the 10 pounds of tributyl phoshite, and the 40 pounds of polyisobutene are then dumped into the kettle 14 and thoroughly

agitated with the aluminum stearate mixture therein.

The lithium stearate mixture in the kettle **10** is now withdrawn through a line **18** and forced by means of a pump therein into the kettle **14** where it is quickly incorporated into the aluminum stearate mixture. The resulting slurry at a temperature of about 150°–200° F. is withdrawn from the kettle **14** through a line **20** and forced by means of a pump therein into the upper portion of a charging kettle **22** which is provided with the usual mixing equipment and a steam jacket for maintaining the temperature at from 150° to 200° F. The kettle **22** is used for supplying the complete mixture of the ingredients to the further processing equipment in a substantially continuous manner. The mixture is withdrawn from the kettle **22** through a line **24** and forced by means of a pump therein into and through a long heating coil **26** of small diameter mounted in an oil bath in a long cylindrical reaction heater **28**. The mixture is pumped under pressure through the small coil **26** and simultaneously heated and worked therein as it is forced through the coil. In the apparatus represented in the drawing the coil **26** comprised 800 feet of ⅜ inch pipe mounted in an oil bath held at a temperature of approximately 440° F. The grease slurry from the kettle **22** was pumped through the coil at a rate of 10 to 15 pounds per minute and heated to a final temperature of about 435° F. One important aspect of the heating is the control of the temperature and the throughput so that the grease slurry is simultaneously heated and worked for approximately three minutes in the temperature range of from about 430° to 435° F.

The grease reaching the end of the outlet of the coil **26** is conducted through a transfer line **30** into a hot receiving tank **32** from which the hot grease is picked up by a chilled steel cylinder **34** and instantly chilled to a temperature of from 70° to 100° F., depending upon the temperature of the available cooling water. The grease congeals on the chilling drum **34**, and during the rotation of the drum it is removed by a scraper **36** and passed by means of a chute **38** into a receiver **40**. Quick and almost instantaneous chilling of the hot grease from the high temperature of about 435° F. to at least 100° F., is extremely important in the production of a stable plastic grease.

The grease discharged from the heating coil **26** is maintained at approximately the final desired temperature right up to the instant of chilling, by providing a jacket **42** around the greater portion of the transfer line **30**, the jacket being filled with the hot oil from the reaction heater **28**. Furthermore, the hot oil from the jacket **42** is used to maintain the temperature of the hot grease in the receiving tank **32**, which is provided with a jacketed bottom **44** as indicated in the drawing. The hot oil is drawn from the jacket **42** through a line **46** into one end of the jacket **44**, withdrawn from the opposite end of the jacket **44** and conducted by means of a return pipe **48** and a pump **50** back into the chamber **28** at the opposite end of the jacket **42**. The chamber **28** (of the shell still type) is provided with the usual fire box mounting **52** and may be heated by oil or gas burners as indicated.

The apparatus as shown may be operated substantially continuously by making up the lithium and aluminum soap mixtures separately in the kettles **10** and **14** and using the large charging kettle **22** to maintain a continuous supply for the coil-heating-and-working operation. The pipe lines **20** and **24** are provided with valves and a bypass line **54** by which the slurry may be supplied to the pump in the line **24** directly from the kettle **14**. It is therefore possible to use the kettles **14** and **22** alternately, and provision may be made for dumping the contents of kettle **10** into the kettle **22**.

The handling of the lithium stearate or other lithium soap is an extremely sensitive matter, and it is extremely important that the grease be finished as soon as reasonably possible after the lithium stearate slurry is mixed with the aluminum stearate slurry. However, the mixing procedure described above may be varied and avoid the use of the kettle **10** by first making up a slurry of all of the ingredients except the lithium stearate in the kettle **14**, and then at the last moment dump in the lithium stearate, thoroughly incorporating it in the slurry and then charge it to the heating and working coil **26** or to the charging kettle **22**.

The chilling drum **34** which is mounted so that it dips in the grease in the container **32** may be rotated by any suitable means such as a drive belt or chain and pulley **56**. Cooling water is supplied to the drum **34** through a line **58** and withdrawn therefrom through a line **60**. The rotation of the drum **34** is maintained sufficiently rapid to chill and remove the grease as fast as it accumulates in the container **32**. The drum dips into the molten grease about 1" and the thickness of the layer after cooling is dependent on the speed of the drum and the temperature of the chilling medium.

The 4000 pounds of lithium base grease produced in accordance with the above example illustrating the invention, had the following properties:

Penetration (ASTM)	270
Water absorption	40%
Melting point	381° F.
Navy beater test	satisfactory
Navy torque test	satisfactory

The manufacture of greases by the process of the present invention poduces a grease which is uniform in composition, stability and in other characteristics, so that it can be sent directly from the receiver **40** to any suitable canning or other packing means. Furthermore, successive batches of any particular grease made in accordance with the process of the present invention will have the same uniform characteristics because it is possible to maintain identical conditions in successive runs. The heating and working coil of small diameter and great length provides an effective means for controlling the heating and working of the grease. While the process is preferably adapted for the manufacture of lithium base greases, it may be used in the manufacture of known greases by the use of predetermined proportions of the desired metal soap or soaps and lubricating oil stocks. Lithium base greases may be made with Pennsylvania lubricating oil stocks by the addition of aluminum soaps as described in the above example or by replacing the aluminum soap with stearic acid. However, the grease made with stearic acid in place of aluminum soap is not water repellent. In the use of aluminum soaps, for example the mono- di- or tri-stearates, they should be dry, and the oils used should be dry in order to make a satisfactory lithium base grease.

In the foregoing example, mention is made of the use of an isobutylene polymer which was used to increase the tackiness and adherent properties of the grease. Other elastic polymers such as rubber latex, gum rubber, "Paratac" a polymer similar to the polyisobutylene, chlorinated rubber, butyl rubber, buna rubber, and other elastic organic compounds may be used. Usually about 0.5% to 1% of such material is satisfactory. The butylene polymer such as polymerized isobutylene may have a molecular weight of from about 8000 to about 25,000. In using "Paratac" the proportion necessary was only about half that of the polyisobutylene used in the illustrative example.

The proportions of lithium stearate and aluminum stearate in the grease may vary somewhat according to the characteristics desired for the final product, but the ratio of aluminum stearate to lithium stearate should be about 2% to 10%. For a dropping point of about 375° F., 11% of lithium stearate and 2.2% of aluminum stearate were found necessary when using the 600 steam refined Pennsylvania lubricating oil. Other lithium and aluminum soaps may be used.

In the manufacture of various types of greases or greases for various purposes, the temperature conditions in the apparatus may be varied. For example, the temperature in the kettle 14 may vary from 250° F. to 300° F., while the oil bath in the cylindrical reaction heater 28 may vary from about 400° F. to 450° F. The final temperature to which the grease is heated in the coil 26 may also vary from about 400 F. to 450° F. and the reaction time changed by varying the throughput. While the invention has been illustrated in connection with a specific example, it will be understood that both the penetration and the melting point of the grease may be varied according to the type of grease desired. A suitable lithium base grease may have a dropping point of from 375° F. to 395° F.

It is to be understood that the invention is not limited to the specific example given but that various conditions such as the reaction time may be changed in accordance with oil stocks and soaps employed, as would be apparent to one skilled in the art.

Having thus described the invention, what is claimed as new is:

1. The method of manufacturing a lithium base grease comprised mostly of a Pennsylvania lubricating oil stock, about 11 per cent by weight of a lithium soap, and about 2.2 per cent by weight of an aluminum soap, which comprises forming a slurry of a substantial part of the lubricating oil and lithium soap at a temperature of about 170° F., separately forming a slurry of the aluminum soap in another portion of the lubricating oil at a temperature of about 270° F., mixing the aluminum soap slurry and the lithium soap slurry and immediately passing the mixture through a long heating coil of relatively small diameter in which the mixture is simultaneously heated and worked, heating the mixture in the final stages of the heating operation for a period of about three minutes in the temperature range of 430° to 440° F., discharging the resulting grease mixture from the heating coil and instantly chilling the grease in very thin layers to a temperature of 100° F. or below to produce a stable homogeneous lithium base grease.

2. The method as defined by claim 1 in which a portion of the lubricating oil is mixed with a small proportion of an oxidation inhibitor and a small proportion of an elastic polymer and in which this mixture is incorporated in the aluminum soap slurry.

3. The method as defined by claim 1 in which the hot grease is discharged from the heating coil into a shallow container and is chilled on a rotating chilling cylinder dipping into the hot grease in the container which progressively picks up thin layers of grease therefrom.

4. The method as defined by claim 1 in which the grease in the heating coil is heated by a bath of oil surrounding the coil and maintained at a temperature of about 445° F.

5. The method of manufacturing a lithium base grease, which comprises forming a slurry of a lithium soap and a Coastal lubricating oil stock in the desired proportions for making a grease, introducing the slurry into a long heating coil of small diameter and forcing the slurry therethrough at a rate adapted to give it a thorough working, simultaneously heating the slurry to a final temperature of 400–450° F. at a rate such that it is held within that range for a maximum period of about 3 minutes, and immediately chilling the mixture to a temperature of 100° F. or lower by picking up thin layers of it on a chilled roll.

6. The method as defined by claim 5 in which the original slurry contains a small proportion of an anti-oxidant and a small proportion of an elastic polymer.

7. The method of manufacturing a lithium base grease, which comprises forming a slurry of an aluminum soap and a lubricating oil stock at a temperature of from 250° to 300° F., forming a second slurry of a lithium soap and a lubricating oil stock at a temperature of from 150° to 200° F., mixing the aluminum soap slurry and lithium soap slurry and passing the resulting mixture through a long heating coil of small diameter in which the mixture is simultaneously worked and progressively raised in temperature to from 400° to 450° F., said heating being carried out at such a rate that the slurry is held within said final temperature range for a maximum of about three minutes, and quickly chilling the grease mixture in very thin layers to a temperature of 100° F. or below after it reaches said temperature of 400° to 450° F.

8. The method as defined by claim 7 in which approximately 11% of lithium soap and 2.2% of aluminum soap are used, the remaining percentage of the mixture being almost entirely lubricating oil stock.

9. The method of manufacturing a stable plastic grease which comprises forming a slurry of metal soaps including lithium soap and a lubricating oil stock in the desired proportions for making a grease, introducing the slurry into a long heating coil of small diameter and forcing the slurry therethrough at a rate adapted to give a thorough working of the slurry while it is simultaneously heated and raised in temperature to the desired final temperature, heating the slurry to a final temperature adapted to produce a stable grease, and within a few minutes after it has reached such final temperature quickly chilling the grease mixture in thin layers to a low temperature of approximately 100° F. or lower.

10. The method of manufacturing a lithium base grease which comprises forming a slurry of an aluminum soap and lubricating oil at a temperature of from 250 to 300° F., separately forming a slurry of a lithium soap and lubricating oil at a temperature of from 150° to 200° F., mixing

the aluminum soap slurry and the lithium soap slurry, heating the mixture to a temperature of from 400° to 450° F. while simultaneously working it, the mixture being held within that temperature range for not more than a few minutes, and then quickly chilling the grease mixture to a temperature of 100° F. or less.

11. The method of manufacturing a lithium base grease which comprises forming a slurry of an aluminum soap and a lubricating oil at a temperature of from 250 to 300° F., separately forming a slurry of a lithum soap and a lubricating oil stock at a temperature of from 150° to 200° F., mixing the aluminum soap slurry and lithium soap slurry, passing the resulting mixture in a confined stream of narrow cross section through a heating zone in which the mixture is rapidly heated to a temperature of from 400 to 450° F., and quickly chilling the grease mixture to a temperature of 100° F. or below within a few minutes after it has reached said temperature of 400° to 450° F.

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